



BAKER BOTTS LLP

Please type a plus sign (+) inside this box → ☐

ERW

| | | |
|--|------------------------|---------------------------|
| TRANSMITTAL FORM <i>(to be used for all correspondence after initial filing)</i> | Application Number | 10/773,870 |
| | Filing Date | February 6, 2004 |
| | First Named Inventor | Trevor Dean |
| | Group Art Unit | 1731 |
| | Examiner Name | Not yet assigned |
| Total Number of Pages in This Submission | Attorney Docket Number | A36142-PCT- (072035.0135) |

| ENCLOSURES (check all that apply) | | |
|---|--|--|
| <input checked="" type="checkbox"/> Fee Transmittal Form <input type="checkbox"/> Fee Attached <input type="checkbox"/> Amendment / Reply <input type="checkbox"/> After Final <input type="checkbox"/> Affidavits/declaration(s) <input type="checkbox"/> Extension of Time Request <input type="checkbox"/> Express Abandonment Request <input type="checkbox"/> Information Disclosure Statement <input checked="" type="checkbox"/> Certified Copy of Priority Document(s) <input type="checkbox"/> Response to Missing Parts/Incomplete Application <input type="checkbox"/> Response to Missing Parts under 37 CFR 1.52 or 1.53 | <input type="checkbox"/> Assignment Papers (for an Application) <input type="checkbox"/> Drawing(s) <input type="checkbox"/> Licensing-related Papers <input type="checkbox"/> Petition <input type="checkbox"/> Petition to Convert to a Provisional Application <input type="checkbox"/> Power of Attorney, Revocation Change of Correspondence Address <input type="checkbox"/> Terminal Disclaimer <input type="checkbox"/> Request for Refund <input type="checkbox"/> CD, Number of CD(s) _____ | <input type="checkbox"/> After Allowance Communication to Group <input type="checkbox"/> Appeal Communication to Board of Appeals and Interferences <input type="checkbox"/> Appeal Communication to Group (Appeal Notice, Brief, Reply Brief) <input type="checkbox"/> Proprietary Information <input type="checkbox"/> Status Letter <input type="checkbox"/> Other Enclosure(s) (please identify below): |
| Remarks <input type="checkbox"/> | | |

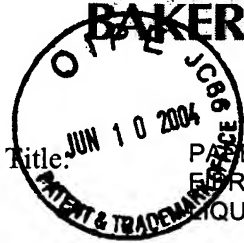
| SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT | |
|--|---|
| Firm or Individual name | BakerBotts LLP 30 Rockefeller Plaza New York, NY 10112 |
| Signature | <i>Rochelle K. Seide</i> Att Name: Rochelle K. Seide PTO Reg: 32,300 |
| Date | June 4, 2004 |

| CERTIFICATE OF MAILING | |
|---|--|
| I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner for Patents, PO Box 1450, Alexandria, VA 22313-1450 on this date: June 4, 2004 | |
| Typed or printed name | Rochelle K. Seide |
| Signature | <i>Rochelle K. Seide</i> Date June 4, 2004 |



BAKER BOTTS LLP

Attorney Docket Number: A36142-PCT- (072035.0135)



Title:

PAPER PLANT - METHOD AND APPARATUS FOR PRODUCING PULP FROM CELLULOSIC
FIBROUS RAW MATERIALS AND RECOVERING CHEMICALS AND ENERGY FROM PULP
LIQUORS

Use Space Below for Additional Information:



THIS PAGE BLANK (USPTO)

BAKER BOTTS LLP



FEE TRANSMITTAL for FY 2003

Effective 10/01/2003. Patent fees are subject to annual revision.

☐ Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT (\$0)

Complete if Known

| | |
|----------------------|---------------------------|
| Application Number | 10/773,870 |
| Filing Date | February 6, 2004 |
| First Named Inventor | Trevor Dean |
| Examiner Name | Not yet assigned |
| Art Unit | 1731 |
| Attorney Docket No. | A36142-PCT- (072035.0135) |

METHOD OF PAYMENT (check all that apply)

☐ Check ☐ Credit card ☐ Money Order ☐ Other ☐ None

☒ Deposit Account:

 Deposit
Account
Number
Deposit
Account
Name

02-4377

Baker Botts LLP

The Commissioner is authorized to: (check all that apply)

☐ Charge fee(s) indicated below ☒ Credit any overpayments

☒ Charge any additional fee required under 37CFR 1.16 and 1.17

☐ Charge fee(s) indicated below, except for the filing fee to the above-identified deposit account.

FEE CALCULATION

1. BASIC FILING FEE

| Large Entity | | Small Entity | | Fee Description | Fee Paid |
|--------------|----------|--------------|----------|------------------------|----------|
| Fee Code | Fee (\$) | Fee Code | Fee (\$) | | |
| 1001 | 770 | 2001 | 385 | Utility filing fee | |
| 1002 | 340 | 2002 | 170 | Design filing fee | |
| 1003 | 530 | 2003 | 265 | Plant filing fee | |
| 1004 | 770 | 2004 | 385 | Reissue filing fee | |
| 1005 | 160 | 2005 | 80 | Provisional filing fee | |
| SUBTOTAL (1) | | | | | (\$0) |

2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE

| | | Extra Claims | | Fee from below | | Fee Paid |
|--------------------|----------------------|--------------|----------------------|----------------|----------------------|----------------------|
| Total Claims | <input type="text"/> | - 20 = | <input type="text"/> | X | <input type="text"/> | <input type="text"/> |
| Independent Claims | <input type="text"/> | - 3 = | <input type="text"/> | X | <input type="text"/> | <input type="text"/> |
| Multiple Dependent | <input type="text"/> | | | | | |

| Large Entity | | Small Entity | | Fee Description |
|--------------|----------|--------------|----------|--|
| Fee Code | Fee (\$) | Fee Code | Fee (\$) | |
| 1202 | 18 | 2202 | 9 | Claims in excess of 20 |
| 1201 | 86 | 2201 | 43 | Independent claims in excess of 3 |
| 1203 | 290 | 2203 | 145 | Multiple dependent claim, if not paid |
| 1204 | 86 | 2204 | 43 | ** Reissue independent claims over original patent |
| 1205 | 18 | 2205 | 9 | ** Reissue claims in excess of 20 and over original patent |

SUBTOTAL (2) (\$0)

**or number previously paid, if greater; For Reissues, see above

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity Small Entity

| Fee Code | Fee (\$) | Fee Code | Fee (\$) | Fee Description | Fee Paid |
|----------|----------|----------|----------|--|----------|
| 1051 | 130 | 2051 | 65 | Surcharge - late filing fee or oath | |
| 1052 | 50 | 2052 | 25 | Surcharge - late provisional filing fee or cover sheet | |
| 1053 | 130 | 1053 | 130 | Non-English specification | |
| 1812 | 2,520 | 1812 | 2,520 | For filing a request for <i>ex parte</i> reexamination | |
| 1804 | 920* | 1804 | 920* | Requesting publication of SIR prior to Examiner action | |
| 1805 | 1,840* | 1805 | 1,840* | Requesting publication of SIR after Examiner action | |
| 1251 | 110 | 2251 | 55 | Extension for reply within first month | |
| 1252 | 420 | 2252 | 210 | Extension for reply within second month | |
| 1253 | 950 | 2253 | 475 | Extension for reply within third month | |
| 1254 | 1,480 | 2254 | 740 | Extension for reply within fourth month | |
| 1255 | 2,010 | 2255 | 1,005 | Extension for reply within fifth month | |
| 1401 | 330 | 2401 | 165 | Notice of Appeal | |
| 1402 | 330 | 2402 | 165 | Filing a brief in support of an appeal | |
| 1403 | 290 | 2403 | 145 | Request for oral hearing | |
| 1451 | 1,510 | 1451 | 1,510 | Petition to institute a public use proceeding | |
| 1452 | 110 | 2452 | 55 | Petition to revive - unavoidable | |
| 1453 | 1,300 | 2453 | 650 | Petition to revive - unintentional | |
| 1501 | 1,330 | 2501 | 665 | Utility issue fee (or reissue) | |
| 1502 | 480 | 2502 | 240 | Design issue fee | |
| 1503 | 630 | 2503 | 315 | Plant issue fee | |
| 1460 | 130 | 1460 | 130 | Petitions to the Commissioner | 0 |
| 1807 | 50 | 1807 | 50 | Processing fee under 37 CFR 1.17(q) | |
| 1806 | 180 | 1806 | 180 | Submission of Information Disclosure Stmt | |
| 8021 | 40 | 8021 | 40 | Recording each patent assignment per property (times number of properties) | |
| 1809 | 770 | 2809 | 385 | Filing a submission after final rejection (37 CFR 1.129(a)) | |
| 1810 | 770 | 2810 | 385 | For each additional invention to be examined (37 CFR 1.129(b)) | |
| 1801 | 770 | 2801 | 385 | Request for Continued Examination (RCE) | |
| 1802 | 900 | 1802 | 900 | Request for expedited examination of a design application | |

Other fee (specify) _____

*Reduced by Basic Filing Fee Paid

SUBTOTAL (3) (\$0)

SUBMITTED BY

Name (Print/Type)

Rochelle K. Seide

Registration No.
(Attorney/Agent)

32,300

(Complete if applicable)

Telephone 212.408.2500

Signature

Rochelle K. Seide

Date

June 4, 2004



THIS PAGE BLANK (USPTO)

THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Trevor Dean

Customer No. : 21003

Serial No. : 10/773,870

Examiner : Not Yet Assigned

Filed : February 6, 2004

Group Art Unit: 1731

For : PAPER PLANT – METHOD AND APPARATUS FOR PRODUCING
PULP FROM CELLULOSIC FIBROUS RAW MATERIALS AND
RECOVERING CHEMICALS AND ENERGY FROM PULP LIQUORS

SUBMISSION OF PRIORITY DOCUMENT

I hereby certify that this paper is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450

June 4, 2004

Date of Deposit

Rochelle K. Seide

Attorney Name

Signature

32,300

PTO Registration No.

June 4, 2004

Date of Signature

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

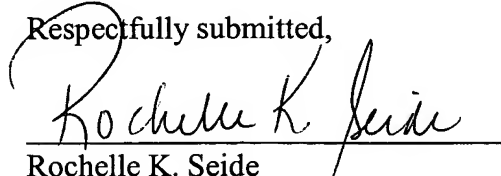
A claim for priority under the provisions of 35 U.S.C. §119 for the above-identified U.S. patent application based upon Great Britain Patent Application No. 0119237.6, filed August 7, 2001, was made in the Patent Application Transmittal dated February 6, 2004, and is hereby again made. A certified copy of the Great Britain priority document is enclosed herewith.



THIS PAGE BLANK (USPTO)

There should be no fee required for this submission. However, if any fee is required, or if any overpayment has been made, the Commissioner is hereby authorized to charge any fees, or credit or any overpayments made, to Deposit Account 02-4377. A duplicate copy of this paper is enclosed.

Respectfully submitted,

A handwritten signature in cursive script, reading "Rochelle K. Seide", written over a horizontal line.

Rochelle K. Seide

Patent Office Reg. No. 32,300

Baker Botts LLP
30 Rockefeller Plaza
New York, NY 10112

Attorney for Applicant
(212) 408-2626

Enclosures

THIS PAGE BLANK (USPTO)



INVESTOR IN PEOPLE

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

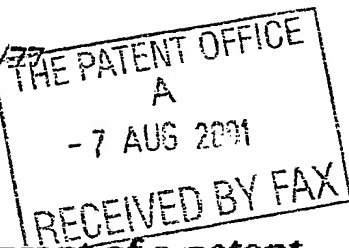
Signed

Dated

27 January 2004

THIS PAGE BLANK (USPTO)

Patents Form 1/77

Patents Act 1977
(Rule 16)The
Patent
Office07AUG01 E551046-1 D02563
P01/7700 0.00-0119237.6**Request for grant of a patent***(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)*

The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference

P895/1/UK

2. Patent application number

(The Patent Office will fill in this part)

07 AUG 2001

0119237.6

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*BioRegional MiniMills (UK) Limited
Sutton Ecology Centre
Honeywood Walk
Carshalton
Surrey SM5 3NSPatents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation

United Kingdom

08204422001

4. Title of the invention

PAPER PLANT

5. Name of your agent *(if you have one)*

Barlin Associates

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*50 Throwley Way
Sutton
Surrey SM1 4BFPatents ADP number *(if you know it)*

0002311001

04046629004

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country

Priority application number
*(if you know it)*Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
*(day / month / year)*8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

YES.

- a) any applicant named in part 3 is not an inventor, or
- b) there is an inventor who is not named as an applicant, or
- c) any named applicant is a corporate body.

See note (d))

Patents Form 1/77

0013580: 07 Aug 01 05:32

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form.
Do not count copies of the same document

Continuation sheets of this form

Description 12

Claim(s) 4

Abstract

Drawing(s) 1

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application

Signature

Barlin Associates

Date

7th August 2001

12. Name and daytime telephone number of person to contact in the United Kingdom

Peter L. Barnes
0208 770 1901

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
b) Write your answers in capital letters using black ink or you may type them.
c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.
e) Once you have filled in the form you must remember to sign and date it.
f) For details of the fee and ways to pay please contact the Patent Office.

- 1 -

PAPER PLANT

This invention relates to a paper plant having a small scale process by which cellulosic raw materials are converted into pulp for papermaking and black liquor effluent generated from the pulping process is treated to recover organic and inorganic chemicals. It is also to be understood that while the combined process has been designed for a paper plant, the individual process steps and apparatus used may be used individually in other suitable processes, not necessarily related to paper production.

Agricultural residues such as wheat and rice straw contain cellulose and can be a good raw material for papermaking. However, as these raw materials are bulky, transportation costs mean that they are best pulped locally and therefore on a relatively small scale of around 10-100 tonnes of pulp production per day.

Pulp mills generate black liquor effluent which if discharged to watercourses causes severe pollution. The technology currently used to treat black liquor effluent is, depending on local economic conditions, only economically viable on a scale of not less than 100-200 tonnes of pulp production per day. The typical scale of a modern wood pulp mill is over 1,000 tonnes of pulp production per day.

Lack of technology to deal with black liquor effluent under 100-200 tonnes per day of production has meant that existing small pulp mills have been forced to close to stop pollution of watercourses. This lack of suitable technology has also prevented the establishment of new small pulp mills, in particular new mills which might have used agricultural residues.

The subsequent lack of demand for small pulp mills has meant that little research and development of small pulp mill technology has been carried out. Consequently small pulp mill technology and straw pulping in particular, has not advanced as far as large scale wood pulping technology has during the latter part of the 20th century. Current small pulp mill technology is therefore relatively resource inefficient. Also there are particular properties of straw as a raw material, which can cause problems in processing and in the quality of the finished pulp which have not been addressed by current technology. These include partially pulped nodes leading to imperfections in the finished paper and drainage problems on the paper machine caused by over processing of the straw fibres. In addition processes currently used to pulp straw

- 2 -

leave silica, which is present in straw, in the black liquor which then forms glass like deposits when the effluent is concentrated for treatment, leading to inefficiencies and down time for cleaning.

The present invention seeks to reduce or obviate one or more of these problems.

According to a first aspect of the invention, there is provided a treatment apparatus for treating elongate cellulosic raw material suitable for use in a paper making plant comprising extracting means for extracting contrary material from the raw material, crushing means for crushing the raw material to remove nodules therefrom and splitting means for splitting the crushed raw material lengthways.

The extracting means may comprise a conveyor belt provided with means for enabling the removal of contrary material.

The crushing means may comprise a pair of counter rotating knurled rollers between which the raw material passes.

The splitting means may comprise a pair of counter-rotating pinned rollers and between which the crushed material passes.

Between the crushing means and the splitting means may be provided means for further removing contrary material present in the crushed material.

According to a second aspect of the invention, there is provided a method for treating raw elongate material suitable for use in a paper making plant comprising extracting contrary material from the raw material, crushing the raw material from which contrary material has been removed to remove nodules therefrom and splitting the crushed raw material lengthways.

The extraction of contrary material may take place on a conveyor belt provided with means for enabling the removal of contrary material.

The crushing of the raw material may take place between a pair of counter rotating knurled rollers between which the raw material passes.

The splitting of the crushed material may take place between a pair of counter

- 3 -

rotating pinned rollers and between which the crushed material passes.

Between the steps of crushing the raw material and splitting the crushed material, further removal of contrary material present in the crushed material may be carried out.

According to a third aspect of the invention, there is provided an apparatus for pulping raw material comprising a counter rotating twin screw conveyor, the conveyor being divided up into a plurality of zones, means for inserting treatment materials into at least one zone and means for controlling the temperature and/or pressure of at least one of the zones.

The conveyor may comprise at least three zones comprising a feed zone, a treatment zone to which treatment material is added and a pressure zone maintained at a pressure above atmospheric.

The conveyor may comprise five zones comprising a feed zone, a treatment zone to which treatment material is added, a first pressure zone at a pressure greater than atmospheric to which treatment material is added, a second pressure zone at a pressure higher than the first pressure zone and a third pressure zone at a lower pressure than the second pressure zone.

The pressure and temperature of the first and third pressure zones may be the same.

Steam may be inserted into the treatment zone and pulping agents may be inserted into the first pressure zone.

The feed zone and the treatment zone may be maintained at atmospheric pressure.

The raw material may be passed through five zones comprising a feed zone, a treatment zone to which treatment material is added, a first pressure zone at a pressure greater than atmospheric to which treatment material is added, a second pressure zone at a pressure higher than the first pressure zone and a third pressure zone at a lower pressure than the second pressure zone.

According to a fourth aspect of the invention, there is provided a method of pulping pre-treated raw material comprising passing the raw material through a plurality of

- 4 -

zones, inserting treatment material into at least one zone and controlling the temperature and/or pressure of at least one of the zones.

The raw material may be passed through at least three zones comprising a feed zone, a treatment zone to which treatment material is added and a pressure zone maintained at a pressure above atmospheric.

The raw material may be passed through five zones comprising a feed zone, a treatment zone to which treatment material is added, a first pressure zone at a pressure greater than atmospheric to which treatment material is added, a second pressure zone at a pressure higher than the first pressure zone and a third pressure zone at a lower pressure than the second pressure zone.

The method may comprise controlling the pressure and temperature of the first and third pressure zones to be the same.

The method may comprise inserting steam into the treatment zone and inserting pulping agents into the first pressure zone.

The method may comprise maintaining the feed zone and the treatment zone at atmospheric pressure.

According to a fifth aspect of the invention, there is provided an apparatus for treatment of black liquor effluent produced in a paper pulp manufacturing plant comprising an evaporator for concentrating the liquor to 45-60% solids, a processing vessel for treating the concentrated liquor at a temperature between 400°C and 600°C, and a closed conveyor for transporting the concentrated liquor from the evaporator to the processing vessel.

The processing vessel may comprise the chamber of a toroidal fluidised bed into which the concentrated black liquor is sprayed, the fluidised bed containing an earth oxide at a ratio of 0.3:1 set up under stoichiometric conditions.

The closed conveyor may be a twin screw conveyor with an earth oxide, the ratio of earth oxide to black liquor being between 0.3:1 and 1.2:1 so that it becomes a granular friable material.

- 5 -

The apparatus may further comprise a toroidal fluidised bed to which the output of the twin screw conveyor is fed under stoichiometric conditions.

The apparatus may include means for chemically converting the material in the fluidised bed into sodium hydroxide and/or sodium carbonate and a gas containing liquids with a combustible component which can be utilised for energy production.

According to a sixth aspect of the invention, there is provided a method of treatment of black liquor produced in a paper manufacturing plant comprising concentrating the liquor to 45-60% solids, passing the concentrated liquor to a processing vessel and treating the concentrated liquor therein at a temperature of between 400°C and 600°C.

The concentrated black liquor may be sprayed into the chamber of a toroidal fluidised bed containing an earth oxide at a ratio of 0.3:1 earth oxide to black liquor dry solids dry solids and set up under stoichiometric conditions.

The concentrated black liquor may be fed into a twin screw conveyor with an earth oxide, the ratio of earth oxide to black liquor being between 0.3:1 and 1.2:1 so that it becomes a granular friable material.

The output of the twin screw conveyor may be fed to a toroidal fluidised bed to under stoichiometric conditions.

The method may include means for chemically converting the material fluidised bed into sodium hydroxide and/or sodium carbonate and a gas containing liquids with a combustible component which can be utilised for energy production.

The invention will now be described in greater detail, by way of example, with reference to the drawings, the single figure of which is a schematic view of a roller arrangement for use in the process of figure 1;

Wheat straw is usually chopped before pulping. Wheat straw contains nodes within the stem which usually remain intact if straw is chopped before pulping. This is a serious drawback in the production of quality paper pulp with the resulting poor quality paper being produced.

- 6 -

In accordance with the invention, a new method is employed which crushes the nodes, opens out the straw stem lengthways in a gentle fashion and feeds the raw material into the pulper in a positive, metered and continuous process.

The entire process will now be described in general terms:-

Firstly the material to be treated is fed to a conveyor belt which has included in it means for the extraction of contrary material from the raw material. The conveyor feeds the resulting raw material to a series of pairs of rollers. A first pair crushes the raw material to remove nodes and a second pair splits the raw material lengthwise. A third pair, between the first and second pairs, removes any contrary material produced in the crushing operation.

Next, the pre treated raw material is fed to a counter rotating twin screw conveyor which is divided into five zones comprising a feed zone, a treatment zone to which treatment material is added, a first pressure zone at a pressure greater than atmospheric to which further treatment material is added, a second pressure zone at a pressure higher than the first pressure zone and a third pressure zone at a lower pressure than the second pressure zone.

The pulped material out put from this conveyor may then be further treated in dependence on the quality of paper to be produced.

Black liquor produced by the paper manufacturing plant is treated in an evaporator for concentrating the liquor to 45-60% solids and passed by a closed conveyor at a temperature above 90°C to a processing vessel where it is processed at a temperature between 400 and 600°C.

The processing vessel is either a toroidal fluidised bed into which the concentrated black liquor is sprayed, the fluidised bed containing an earth oxide at a ratio of 0.3:1 earth oxide to black liquor solids or a twin screw conveyor together with an earth oxide in a ratio of earth oxide to black liquor solids of between 0.3:1 and 1.2:1. These processes convert the material into sodium hydroxide and/or sodium carbonate and a gas containing liquids with a combustible component which can be used for energy production.

The individual parts of the process will now be described:-

- 7 -

Referring now to Figure 2, after the bales of straw have been opened, straw is passed along a conveyor belt 101 where heavy items such as stones and other contraries such as plastic string are removed. The straw is then passed in to a feed hopper 103 which feeds the straw into to an arrangement of knurled rollers 105 and 107 which crush the nodes in the straw stem and rollers with pins which open the straw stem out lengthways in a gentle fashion. Thus straw is fed between first and second counter-rotating knurled crushing rollers 105 and 107 to crush the straw nodes. The crushed material then passes through two counter-rotating intermediate rollers 109 and 111 which prevent any contrary materials from damaging the rollers below.

The straw then passes through two more rollers 113 and 115, this time rotating in the same direction. These latter rollers are provided with pins which open and shred the straw lengthways and act in co-operation with a feed shoe.

The action of this system leaves the straw as shortened and opened out/ shredded material without nodes. This will facilitate quicker chemical and steam penetration and so faster and more uniform pulping, whilst treating the fibres gently so preserving their length. This results in the production of an improved quality of pulp. Including a 70% reduction in visible "shiners" in the paper sheet, due to dispersion of parenchyma cells, improved drainage, a higher tensile and tear strength, a higher pulp yield and a reduced demand for pulping chemicals.

The treated straw then drops from the pinned rollers 113 and 115 into a feed hopper 117 leading to either a conveyor or blower system (not shown) which feeds the treated straw into a live bottom bin for buffer storage of the prepared material prior to pulping.

The pinned rollers are self-cleaning when used with longer fibred cellulosic raw materials such as hemp and flax. This is to prevent the material wrapping around the rollers and fouling the apparatus.

The above discussed pinned and knurled or fluted roller opening and feeding system is specifically designed for straw but, with minor modifications, could be used for any other suitable raw materials including flax, hemp, bagasse and wood.

- 8 -

The above described process has been tested and developed with straw, flax and hemp through pilot scale laboratory trials.

The raw material from the buffer storage is thereafter pulped. To this end, the raw material (straw, flax, hemp, bagasse, wood chips or any other cellulosic raw material) is drawn into a specially designed co-rotating twin screw extrusion unit. In this unit the screw profiles are specially designed. The screw flights are manufactured from hardened steel with a deep cut flight and are specially designed to minimise fibre damage. This particular design results in a reduced energy demand, which means that a smaller drive shaft and gearbox can be used, which also reduces capital cost. The design of the screw profile and the reduced drive shaft size also allows throughput of raw material to be increased by an anticipated 400% over conventional co-rotating twin screws.

The unit is of modular construction which facilitates making changes to both screw and barrel configurations. This could be a very cost-effective way to make use of one standard twin screw unit to process many different types of cellulosic raw materials and/or to produce different grades of pulp simply by changing the screw and barrel configurations. Machine speeds of between 50-500 rpm could be used. A speed of 50-250 rpm has been used in practice. The speed needs to be adjusted for the raw material used and the pulp quality required.

The twin screw can be built in such a way that chemicals and liquids can be injected and liquids or steam can be vented or removed in each zone. This is a standard feature of twin screw extruders.

[It has further been discovered that a sophisticated gearbox and drive of the type conventionally used in twin screw extruders is not necessary to suitably pulp fibres. A simple gearbox and drive can be used, reducing the capital cost and energy consumption. It is anticipated that the pulping system will consume less than half the energy of a conventional twin screw used for this purpose.

One method of pulping cellulosic raw materials such as straw using the new system is as follows. Using a co-rotating twin screw with a barrel size of 100 millimetres, the co-rotating intermeshing twin screw extruder is set up with five zones as described below.

- 9 -

| Zone | 1 | 2 | 3 | 4 | 5 |
|----------------|-----------|----------------------------------|---|---------------|---------------|
| Action of zone | Feed zone | Steam zone Introduce steam | Pressure zone Introduce NaOH + other pulping agents | Pressure zone | Pressure zone |
| Temp °C | 65 | 100 | 130 | 150 | 130 |
| Pressure (bar) | 0 | 0 | 2-3 | 4-5 | 2-3 |

Cellulosic raw material such as straw is positively fed into Zone 1 using an Auger.

Zone 1 is designed to be as open as possible in order to accept the material into the unit. The temperature is 65 °C in Zone 1.

In zone 2 saturated steam is introduced to prepare the material for pulping. Temperature increases to 100 °C

In Zone 3 temperature and pressure are raised to 130 °C, 2-3 bar pressure. Sodium hydroxide is added at a rate of 12-14% to dry raw material using a 15% solution. Other materials may be added here as will be referred to hereafter

In Zone 4 temperature and pressure are increased to 150 °C and 4-5 bar pressure.

In Zone 5 temperature and pressure are reduced to 130 °C and 2-3 bar pressure in preparation for the material leaving the twin screw system. The material travels through the twin screw unit in between 2-3 minutes. The screw speed is around 200 rpm.

The pulp exiting from the twin screw at this point would have 50% moisture content and would be expected to have a Kappa Number of 50. This is a semi-chemical pulp suitable for use in fluted packaging for example. This result is a function of the rpm and the flight design or time spent in the twin screw extruder

To go on and produce a full chemical pulp it will then be necessary for the pulp to be further digested in a single screw pulper using steam at 1-2 bar pressure (120 °C) for a further 20-40 minutes. A Kappa Number of 14-20 is achieved after this further processing. The pulp is then ready to be bleached using conventional methods.

-10-

The invention also provides a method of precipitating silica present in straw onto cellulosic fibres when pulping straw to make paper and so prevent it entering the black liquor effluent and causing scaling of evaporators or chemical recovery system.

Calcium hydroxide is added in Zone 3 of the twin screw extruder at a rate of 4% to dry raw material (straw) with 8% sodium hydroxide when pulping straw as described previously. This method could be used in any alkaline based pulping system. This has the effect of precipitating sodium silicate onto the cellulosic fibres as calcium silicate. This prevents silica from entering the black liquor effluent and causing scaling of the evaporators or chemical recovery system. --

The above system in common with all traditional chemical pulping methods produces black liquor which must be treated if environmental and health hazards are to be avoided. The present invention provides a treatment process to recover organic and inorganic chemicals from black liquor effluent arising from the pulping of cellulosic raw materials to make paper. It is specifically intended to be used with the above described pulping process but could be used alone to treat black liquor from other pulping processes.

Black liquor effluent arising from the pulping process is collected in a digestion liquor storage tank and concentrated to 45-60% solids using a standard evaporator designed for concentration purposes. Using an enclosed twin screw transport system in order to reduce loss of organic chemicals through vaporisation the concentrated black liquor moved to a processing vessel at a temperature of above 90°C. This temperature is required as black liquor will flow and is easier to work with at this temperature. The black liquor is treated in either of two methods.

In a first method, the black liquor is introduced into a toroidal fluidised bed by spraying the liquor into the chamber of the toroidal fluidised bed in which an inert toroidal fluidised bed containing earth oxide such as lime at a ratio of 0.3:1 of lime to black liquor dry solids has been set up under stoichiometric conditions.

In a second method, the liquor is pre-mixed in the twin screw conveyor with an earth oxide such as lime (CaO) : black liquor dry solids 0.3:1 to become a granular friable material which is then screw fed into a Toroidal fluidised bed under stoichiometric conditions.

- 11 -

In a variation of the second method, the ratio of CaO (lime) to black liquor dry solids may be 1.2:1

In both cases, the chamber of the toroidal fluidised bed is maintained at a temperature range) at between 400-800 °C where the necessary chemical reaction takes place in the space of seconds.

The material is converted by a chemical reaction to;

1. sodium hydroxide and sodium carbonate and lime within the fluidised bed. The bed will overflow through a central point and is then dissolved to recover sodium hydroxide as green liquor in the traditional manner known as re-causticisation. The green liquor is then filtered to make a calcium carbonate sludge and white liquor (containing sodium hydroxide) for re-use in the pulping process.

2. a gas containing liquids with a combustible component which can be utilised for energy production. The gas is collected to power a boiler which will produce energy for use in the pulp mill process line.

The calcium carbonate sludge is dried to remove some water and sent to a second Toroidal fluidised bed reactor such as the Torbed (GB patent no 0068853). At a temperature of 1100°C where calcium carbonate CaCO_3 is converted back to calcium oxide CaO for re-use in the black liquor chemical recovery process.

10% of the fluidised bed material generated will need to be removed from the process continuously in order to prevent the build up of heavy metals and other materials in the loop. This material could be used at a cement factory or brickworks.

If required black liquor below 45% solids can also be processed using this method (and has been tested). However, energy consumption is greater and so this is not the preferred method.

1. It will be appreciated that individual elements of the above described process can be replaced by suitable equivalents without departing from the scope of the invention. Also any of the individual processes and apparatus may be used individually in other processes where they are suitable, not necessarily related to

-12-

paper making.

-13-

2. CLAIMS:-

1. A treatment apparatus for treating raw elongate material suitable for use in a paper making plant comprising extracting means for extracting contrary material from the raw material, crushing means for crushing the raw material to remove nodules therefrom and splitting means for splitting the crushed raw material lengthways.
2. An apparatus as claimed in claim 1, wherein the extracting means comprises a conveyor belt provided with means for enabling the removal of contrary material.
3. An apparatus as claimed in claim 1 or 2, wherein the crushing means comprises a pair of counter rotating knurled rollers between which the raw material passes.
4. An apparatus as claimed in claim 1, 2 or 3, wherein the splitting means comprises a pair of counter rotating pinned rollers rotating in the and between which the crushed material passes.
5. An apparatus as claimed in any preceding claim wherein, between the crushing means and the splitting means are provided means for further removing contrary material present in the crushed material.
6. A method for treating raw elongate material suitable for use in a paper making plant comprising extracting contrary material from the raw material, crushing the raw material from which contrary material has been removed to remove nodules therefrom and splitting the crushed raw material lengthways.
7. A method as claimed in claim 6, wherein the extraction of contrary material takes place on a conveyor belt provided with means for enabling the removal of contrary material.
8. A method as claimed in claim 6 or 7, wherein the crushing of the raw material takes place between a pair of counter rotating knurled rollers between which the raw material passes.

-14-

9. A method as claimed in claim 6, 7 or 8, wherein the splitting of the crushed material takes place between a pair of counter rotating pinned rollers and between which the crushed material passes.
10. An apparatus as claimed in any one of claims 6 to 9, wherein, between the steps of crushing the raw material and splitting the crushed material, further removal of contrary material present in the crushed material is carried out.
11. An apparatus for pulping raw material comprising a counter rotating twin screw conveyor, the conveyor being divided up into a plurality of zones, means for inserting treatment materials into at least one zone and means for controlling the temperature and/or pressure of at least one of the zones.
12. An apparatus as claimed in claim 11, wherein the conveyor comprises at least three zones comprising a feed zone, a treatment zone to which treatment material is added and a pressure zone maintained at a pressure above atmospheric.
13. An apparatus as claimed in claim 11, wherein the conveyor comprises five zones comprising a feed zone, a treatment zone to which treatment material is added, a first pressure zone at a pressure greater than atmospheric to which treatment material is added, a second pressure zone at a pressure higher than the first pressure zone and a third pressure zone at a lower pressure than the second pressure zone.
14. An apparatus as claimed in claim 13, wherein the pressure and temperature of the first and third pressure zones are the same.
15. An apparatus as claimed in claim 13 or 14, wherein steam is inserted into the treatment zone and pulping agents are inserted into the first pressure zone.
16. An apparatus as claimed in claim 13, 14 or 15, wherein the feed zone and the treatment zone are maintained at atmospheric pressure.
17. An apparatus as claimed in any one of claims 13 to 16, wherein calcium hydroxide is inserted into the first pressure zone.
18. A method of pulping pre-treated raw material comprising passing the raw

-15-

material through a plurality of zones, inserting treatment material into at least one zone and controlling the temperature and/or pressure of at least one of the zones.

19. A method as claimed in claim 18, wherein the raw material is passed through at least three zones comprising a feed zone, a treatment zone to which treatment material is added and a pressure zone maintained at a pressure above atmospheric.

20. A method as claimed in claim 18, wherein the raw material is passed through five zones comprising a feed zone, a treatment zone to which treatment material is added, a first pressure zone at a pressure greater than atmospheric to which treatment material is added, a second pressure zone at a pressure higher than the first pressure zone and a third pressure zone at a lower pressure than the second pressure zone.

21. A method as claimed in claim 20, and comprising controlling the pressure and temperature of the first and third pressure zones to be the same.

22. A method as claimed in claim 20 or 21, and comprising inserting steam into the treatment zone and inserting pulping agents into the first pressure zone.

23. A method as claimed in claim 20, 21 or 22, and comprising maintaining the feed zone and the treatment zone at atmospheric pressure.

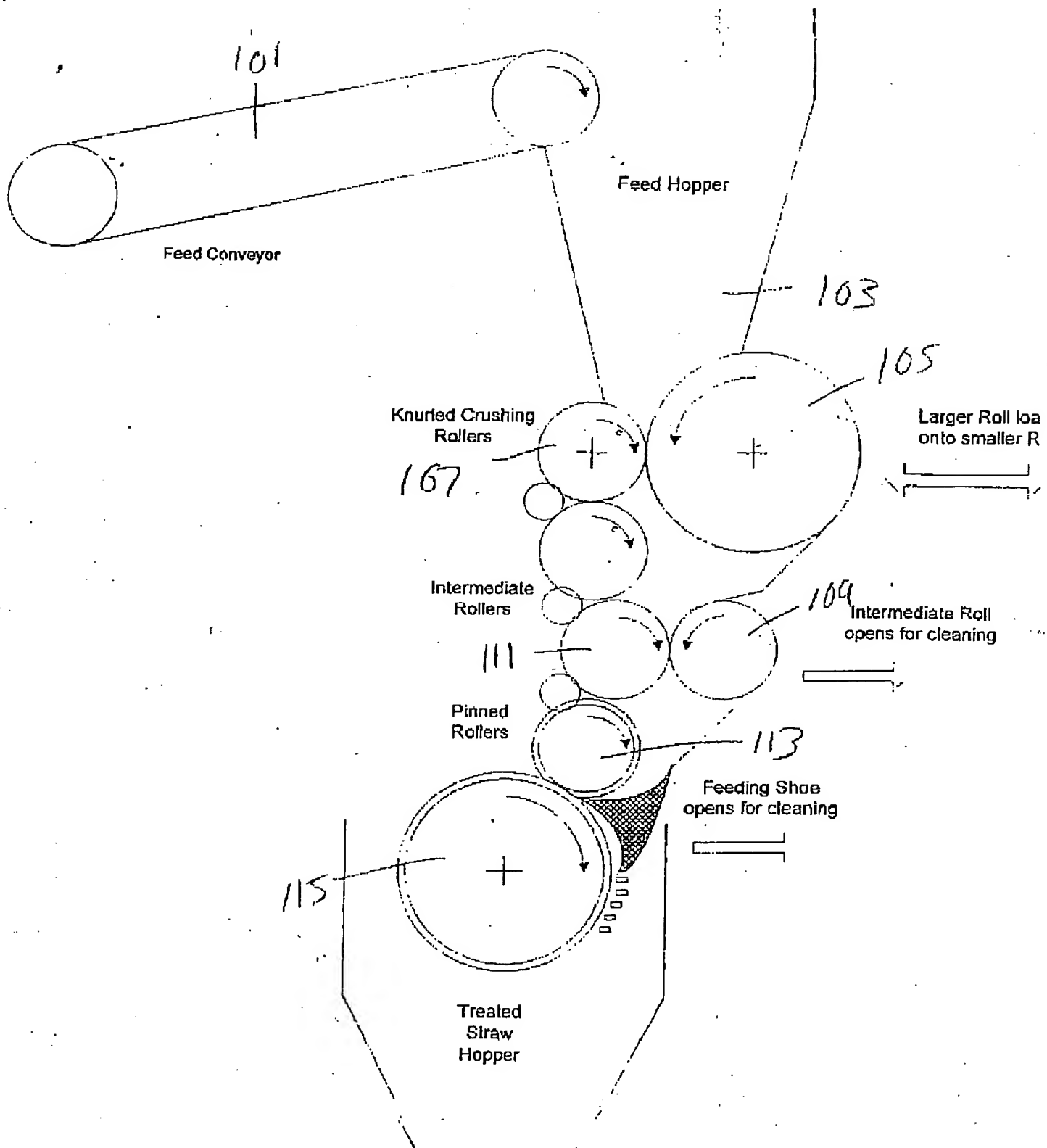
24. A method as claimed in any one of claims 20 to 23, wherein calcium hydroxide is added to the first pressure zone.

25. An apparatus for treatment of black liquor effluent produced in a paper manufacturing plant comprising an evaporator for concentrating the liquor to 45-60% solids, a processing vessel for treating the concentrated liquor at a temperature of between 400-600°, and a closed conveyor for transporting the concentrated liquor from the evaporator to the processing vessel at above 90°C.

26. An apparatus as claimed in claim 25, wherein the processing vessel comprises the chamber of a toroidal fluidised bed into which the concentrated black liquor is sprayed, the fluidised bed containing an earth oxide at a ratio of 0.3:1 set up under stoichiometric conditions.

-16-

27. An apparatus as claimed in claim 26, wherein the closed conveyor is a twin screw conveyor with an earth oxide, the ratio of earth oxide to black liquor being between 0.3:1 and 1.2:1 so that it becomes a granular friable material.
28. An apparatus as claimed in claim 27, wherein the apparatus further comprises a toroidal fluidised bed to which the output of the twin screw conveyor is fed under stoichiometric conditions.
29. An apparatus as claimed in claim 24 or 26 including means for chemically converting the material in the fluidised bed into sodium hydroxide and/or sodium carbonate and a gas containing liquids with a combustible component which can be utilised for energy production.
30. A method of treatment of black liquor produced in a paper manufacturing plant comprising concentrating the liquor to 45-60% solids, passing the concentrated liquor to a processing vessel and treating the concentrated liquor therein at a temperature of between 400-600°C.
29. A method as claimed in claim 28, including spraying the concentrated black liquor into the chamber of a toroidal fluidised bed containing an earth oxide at a ratio of 0.3:1 earth oxide to black liquor dry solids and set up under stoichiometric conditions.
30. A method as claimed in claim 28, wherein the concentrated black liquor is fed into a twin screw conveyor with an earth oxide, the ratio of earth oxide to black liquor dry solids being between 0.3:1 and 1.2:1 so that it becomes a granular friable material.
31. An apparatus as claimed in claim 30, wherein the output of the twin screw conveyor is fed to a toroidal fluidised bed to under stoichiometric conditions.
32. A method as claimed in claim 29 or 31, and including means for chemically converting the material in the fluidised bed into sodium hydroxide and/or sodium carbonate and a gas containing liquids with a combustible component which can be utilised for energy production.



THIS PAGE BLANK (USPTO)